

WL-TM-96-4092



**WEAR AND FRICTION
CHARACTERISTICS OF SELF-
LUBRICATING COPPER -
INTERCALATED GRAPHITE COMPOSITES**

**A. W. Ruff, M. B. Peterson,
A. Gangopadhyay, E. Whitenton**

1989

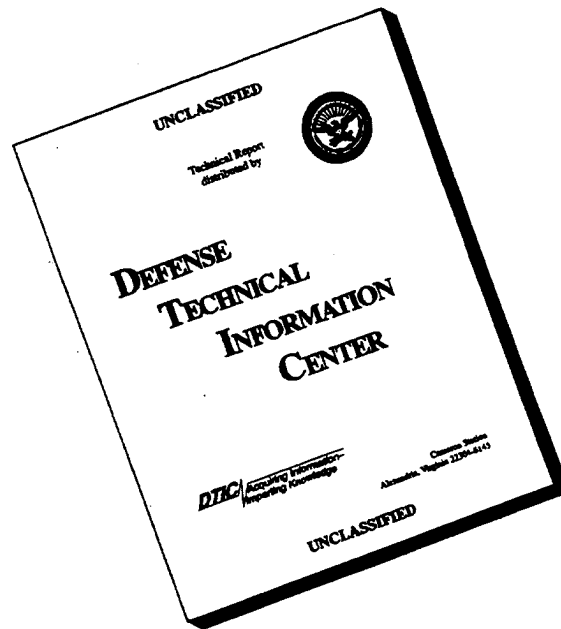
THIS DOCUMENT CONTAINS NEITHER
RECOMMENDATIONS NOR CONCLUSIONS
OF THE AIR FORCE MATERIEL COMMAND

Approved for public release; distribution unlimited

**MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7734**

19960911 024

DISCLAIMER NOTICE



THIS DOCUMENT IS BEST QUALITY AVAILABLE. THE COPY FURNISHED TO DTIC CONTAINED A SIGNIFICANT NUMBER OF PAGES WHICH DO NOT REPRODUCE LEGIBLY.

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE 1989		3. REPORT TYPE AND DATES COVERED
4. TITLE AND SUBTITLE WEAR AND FRICTION CHARACTERISTICS OF SELF-LUBRICATING COPPER - INTERCALATED CRAPHITE COMPOSITE			5. FUNDING NUMBERS	
6. AUTHOR(S) A. W. Ruff, M. B. Peterson, A. Gangopadhyay, and E. Whitenton				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Materials Directorate Wright Laboratory Air Force Materiel Command Wright-Patterson AFB OH 45433-7734			10. SPONSORING / MONITORING AGENCY REPORT NUMBER WL-TM-96-4092	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION / AVAILABILITY STATEMENT APPROVED FOR PUBLIC RELEASE; DISTRIBUTION IS UNLIMITED			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Composite materials consisting of copper metal- matrices with a solid lubricant phase of intercalated (NiCl ₂) graphite have been prepared and studied in sliding wear against type 440C stainless steel at normal temperatures in air. Results on the controlling wear and friction mechanisms in these materials are presented. Beneficial effects were found up to about 15 volume percent intercalated graphite. An analytical model has been developed that relates composite wear to mechanical and tribological properties of the different solid phases in the composite and the interface film.				
14. SUBJECT TERMS			15. NUMBER OF PAGES 8	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT SAR	

GENERAL INSTRUCTIONS FOR COMPLETING SF 298

The Report Documentation Page (RDP) is used in announcing and cataloging reports. It is important that this information be consistent with the rest of the report, particularly the cover and title page. Instructions for filling in each block of the form follow. It is important to *stay within the lines* to meet *optical scanning requirements*.

Block 1. Agency Use Only (Leave blank).

Block 2. Report Date. Full publication date including day, month, and year, if available (e.g. 1 Jan 88). Must cite at least the year.

Block 3. Type of Report and Dates Covered. State whether report is interim, final, etc. If applicable, enter inclusive report dates (e.g. 10 Jun 87 - 30 Jun 88).

Block 4. Title and Subtitle. A title is taken from the part of the report that provides the most meaningful and complete information. When a report is prepared in more than one volume, repeat the primary title, add volume number, and include subtitle for the specific volume. On classified documents enter the title classification in parentheses.

Block 5. Funding Numbers. To include contract and grant numbers; may include program element number(s), project number(s), task number(s), and work unit number(s). Use the following labels:

C - Contract	PR - Project
G - Grant	TA - Task
PE - Program Element	WU - Work Unit Accession No.

Block 6. Author(s). Name(s) of person(s) responsible for writing the report, performing the research, or credited with the content of the report. If editor or compiler, this should follow the name(s).

Block 7. Performing Organization Name(s) and Address(es). Self-explanatory.

Block 8. Performing Organization Report Number. Enter the unique alphanumeric report number(s) assigned by the organization performing the report.

Block 9. Sponsoring/Monitoring Agency Name(s) and Address(es). Self-explanatory.

Block 10. Sponsoring/Monitoring Agency Report Number. (If known)

Block 11. Supplementary Notes. Enter information not included elsewhere such as: Prepared in cooperation with...; Trans. of...; To be published in.... When a report is revised, include a statement whether the new report supersedes or supplements the older report.

Block 12a. Distribution/Availability Statement. Denotes public availability or limitations. Cite any availability to the public. Enter additional limitations or special markings in all capitals (e.g. NOFORN, REL, ITAR).

DOD - See DoDD 5230.24, "Distribution Statements on Technical Documents."

DOE - See authorities.

NASA - See Handbook NHB 2200.2.

NTIS - Leave blank.

Block 12b. Distribution Code.

DOD - Leave blank.

DOE - Enter DOE distribution categories from the Standard Distribution for Unclassified Scientific and Technical Reports.

NASA - Leave blank.

NTIS - Leave blank.

Block 13. Abstract. Include a brief (*Maximum 200 words*) factual summary of the most significant information contained in the report.

Block 14. Subject Terms. Keywords or phrases identifying major subjects in the report.

Block 15. Number of Pages. Enter the total number of pages.

Block 16. Price Code. Enter appropriate price code (*NTIS only*).

Blocks 17. - 19. Security Classifications. Self-explanatory. Enter U.S. Security Classification in accordance with U.S. Security Regulations (i.e., UNCLASSIFIED). If form contains classified information, stamp classification on the top and bottom of the page.

Block 20. Limitation of Abstract. This block must be completed to assign a limitation to the abstract. Enter either UL (unlimited) or SAR (same as report). An entry in this block is necessary if the abstract is to be limited. If blank, the abstract is assumed to be unlimited.

WEAR AND FRICTION CHARACTERISTICS OF SELF-LUBRICATING COPPER - INTERCALATED GRAPHITE COMPOSITES

A.W. Ruff, M.B. Peterson, A. Gangopadhyay,
and E. Whiteman*

Composite materials consisting of copper metal- matrices with a solid lubricant phase of intercalated (NiCl_2) graphite have been prepared and studied in sliding wear against type 440C stainless steel at normal temperatures in air. Results on the controlling wear and friction mechanisms in these materials are presented. Beneficial effects were found up to about 15 volume percent intercalated graphite. An analytical model has been developed that relates composite wear to mechanical and tribological properties of the different solid phases in the composite and the interface film.

1 INTRODUCTION

Self-lubricating materials offer attractive options for many tribological applications where liquid lubrication is not feasible, where thin solid film lubrication is not durable, and where long, unattended operating life is required. The use of solid lubricants as contrasted to liquid lubricants offers broad application potential¹ including higher temperatures, vacuum environments, sealed systems, and long storage life. The present work is part of a program examining basic issues of tribological and mechanical performance in solid, multi-phase, self-lubricating composites. This report discusses results on a metal-matrix (copper) composite using intercalated (NiCl_2) graphite² (referred to hereafter as IG) as the solid lubricating phase. Previous research³ on metal-matrix and polymer-matrix composites with other solid lubricating phases has shown beneficial effects.

2 EXPERIMENTAL APPROACH

Sliding wear tests were carried out using two configurations of a pin-on-ring tribological test system. One configuration permitted loading two pins simultaneously against a rotating steel ring; the other utilized a holder carrying a single pin (Fig.1). The rings were conventional tapered bearing cups, 35 mm diameter, 8 mm wide, type 440C stainless steel, hardness HRC 58, surface finish 0.15 μm RMS. Tribological tests were carried out in laboratory air at normal ambient temperature. Ambient

*Materials Science and Engineering Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899, USA

relative humidity ranged from 10% to 40%. The load was 33 N, sliding speed 0.14 m/s, sliding distance usually 790 m. Specimen pins of 6.25 mm diameter were fabricated by several methods. Compaction of powders was done in a hydraulic press using a die at forces of 5,000 to 10,000 lbf (22 to 44 kN). Pure compacts of copper and of IG were made for the 2-pin tests. Compacts of pre-mixed powder combinations of copper and IG were made for the single pin tests. Figure 2 shows an example of the microstructure of such a mixed compact. Two types of manufactured composites were also used: one containing a series of holes in an hexagonal arrangement with different hole diameters and separations, and the other contained a central slit with different widths (Fig. 3). These

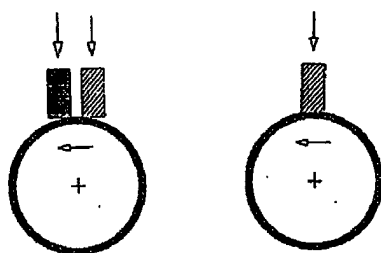


Fig. 1. Schematic of wear geometry using either one or two specimen pins loaded against a rotating ring.



Fig. 2. SEM photograph of Cu-57 vol.% IG powder compacted specimen.

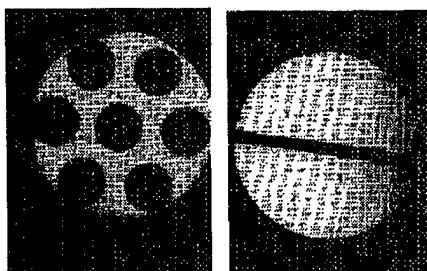


Fig. 3. Optical photographs of manufactured Cu - IG composite specimens. Hole size, hole separation, and slit width were varied.

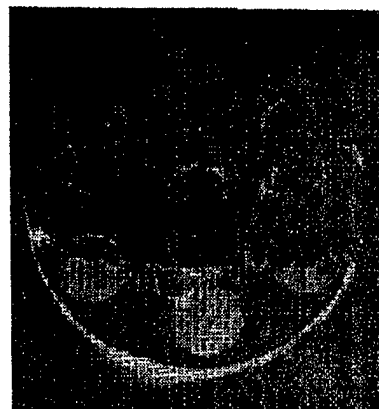


Fig. 4. Wear scar on manufactured Cu - IG composite pin, 6.25 mm diameter.

size variations permitted different effective area and volume percentages of IG and copper under controlled phase shape and spacing.

3 RESULTS AND DISCUSSION

The overall appearance of a typical wear scar on a pin is shown in Fig. 4 for a manufactured composite of Cu-IG. Pin wear volumes were calculated from the pin wear scar dimensions using numeric integration methods. Wear on the rotating ring counterface was not appreciable, although there was some small contribution to the wear debris produced. Figure 5 shows a higher magnification view within the wear scar on a powder composite specimen. Figure 6 shows more detail of the worn surface morphology near the IG phase region in a manufactured composite pin. In both types of specimens, wear led to the establishment of an interfacial film, to the generation of wear debris particles some of which remained in the contact gap, and to surface recession at the location of the soft IG phase. Each of these characteristic features is important in determining the wear and friction mechanisms involved in the tribology of self-lubricating composites.

The interfacial film formed during sliding wear was found on both the composite pin and on the steel ring. While patchy in its distribution and non-uniform in thickness, it is clear that this film is a principle factor controlling wear and friction behavior in this type of system. The interfacial film is comprised of system wear products, namely, IG, copper, and steel (probably Fe, Cr, Ni oxides), based on x-ray microanalysis of the film. Figure 7 shows an SEM photograph from an area on the sliding surface of a manufactured composite pin, near an IG phase region. Sliding is from right to left in this case, and the film being formed on the pin at the exit side of the IG phase region can be clearly seen. X-ray spectra from two areas (marked A and B) are shown. In both spectra,

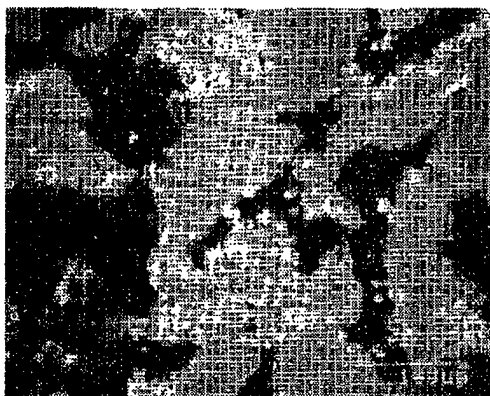


Fig. 5. SEM photograph of worn area in Cu-57 vol.% IG powder compacted specimen.



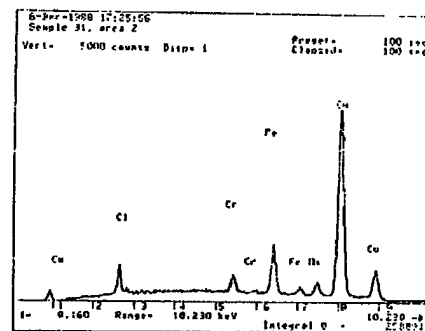
Fig. 6. SEM photograph of worn area on manufactured Cu-25 vol.% IG composite specimen. Sliding direction downward.

the Cu peak originates from wear products in the film, as do the Fe, Cr, and Ni peaks. The Cl peak can be used to determine the amount of IG present (coming from the NiCl_2 intercalation). At thicker regions of the film such as at B, we find proportionally greater amounts of IG (denoted by the Cl peak), and of ring wear debris. Wear debris was not found imbedded into the soft IG phase region. Similar morphology and composition evidence to this has been found in the particulate composite specimens, such as shown in Fig. 5.

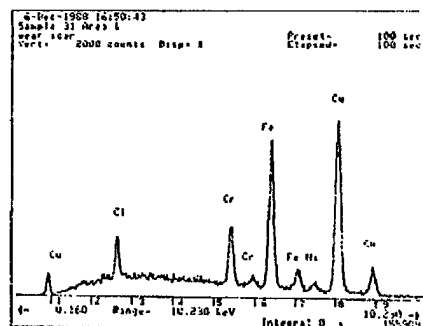
Measured friction and wear data on both types of composites are shown in Figs. 8 and 9. In each case 3 to 5 individual tests were conducted for each of the indicated values of volume % (or area %) IG; average values and a range of \pm one standard deviation are shown. In Fig. 8 the dashed line shows the measured friction value for the 2-pin tests of copper and IG; this result is taken as a baseline for friction since the separate IG pin can supply as much material as needed according to its wear rate. It is seen that friction decreases from a value of about 0.5 for copper to values about 0.15 as the IG fraction in the composite increases. Most of the beneficial decrease occurs up to about 15 vol. % IG. The wear rate for the composites is shown in Fig. 9a. The dashed line again refers to the wear of a Cu pin in 2-pin tests. A reduction in wear occurs on adding up to about 15 vol. % IG. Higher amounts of IG lead to increased wear. This type of behavior suggests that two processes are involved. It is thought that since higher amounts of IG are associated with lower copper content in the composite, that mechanical weakening in the supporting copper matrix takes place at higher levels of IG content and this leads to increased wear. If true, then normalization of the wear data by the



a



b



c

Fig. 7. (a) SEM photograph of worn area in Cu-25 vol.% IG composite. Sliding direction is right to left. Wear debris is collected at entrance to the recessed IG phase region. Interface film formed at exit of IG phase region. (b) X-ray microanalysis from interface film at location A and (c) location B show proportion of IG, Cu, and Fe, Cr, Ni from steel counterface.

vol. % (or area %) Cu in each composite should remove the influence on the wear data of the matrix strength. This is equivalent to applying a rule-of-mixtures for the composite strength influence. Figure 9b shows the normalized wear rate data obtained by this method. It is seen that a monotonic decrease in normalized wear occurs with increasing IG fraction, and that most of the beneficial effect is accomplished by adding up to 15 vol. % IG. This decrease with increasing fraction of IG is consistent with the wear model described next.

4 WEAR MODEL

The steady state thickness of the IG interfacial film is affected by (1) growth as IG is worn away from the composite and added to the film, and (2) shrinkage as the film itself is worn away. Therefore

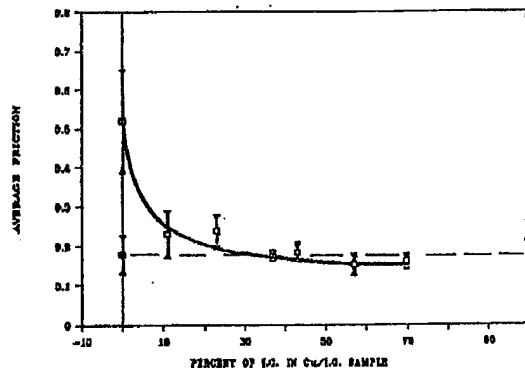
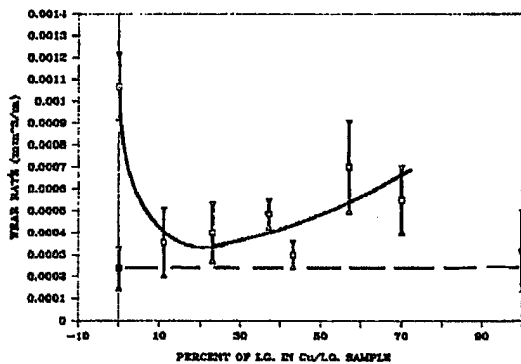
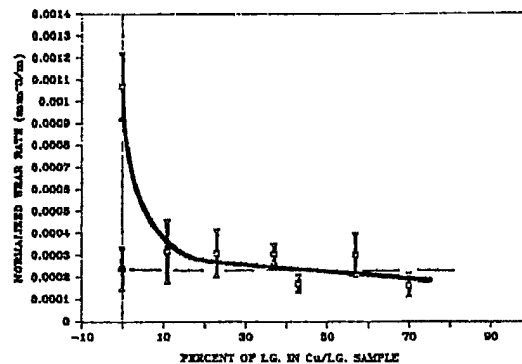


Fig. 8. Friction coefficient results for the Cu - IG composites. Dashed line represents baseline for 2-pin tests. Range of \pm std. dev. shown for each average value.



a



b

Fig. 9. (a) Wear rate results for Cu - IG composites. Dashed line represents wear rate baseline from 2-pin tests. (b) Same wear rate data normalized to volume (or area) fraction of Cu in the composite (in the wear scar area).

the wear volume per unit sliding distance per unit contact pressure for both the film (f) and the lubricant phase (lp) in the composite, can be expressed using the Archard-Holm wear equation as:

$$K_{lp} A_{lp} / H_{lp} \geq K_f A_f / H_f \quad (1)$$

where K is the wear coefficient, H the indentation hardness, and A the contact area of the lubricant phase and the interfacial film.

But $A_f = A_{\text{contact}}$; $H_{lp} = H_c(\text{composite})$; $K_{lp} = K_c(\text{composite})$

since the lubricant phase is part of the composite, as far as the load distribution in the contact region and wear are concerned.

Then $K_c A_{lp} / H_c \geq K_f A_{\text{contact}} / H_f.$ (2)

Thus, it is required for the wear coefficient of the composite if the interfacial film is to maintain a steady-state thickness:

$$K_c \geq K_f (H_c / H_f) (\text{lubricant fraction in composite})^{-1} \quad (3)$$

The observed decrease in wear rate (and thus wear coefficient) for the composite shown in Fig. 9 with increasing graphite fraction is consistent with Eqn. 3. This relation can be further refined to include composite micro-structural parameters such as phase diameter and mean-free-path.

5 CONCLUSIONS

Intercalated (NiCl_2) graphite in copper provides a lubricating, wear-reducing film when worn against 440C steel. Friction coefficients can be as low as 0.15 in air. Volume wear rate decreased by a factor of 5x with a full IG film present in the contact, and by a factor of 4x with a composite specimen containing 12 vol. % IG in copper. Increasing IG content above 15 vol. % leads to increasing wear due to loss of composite strength. Composite worn surface morphology shows three significant mechanisms: (i) lubricating interfacial film formation at the exit region of the soft phase, (ii) recession from the average surface at the soft phase locations, (iii) collection of wear debris in the recess at the entrance region, thus removing some of the wear debris but blocking part of the soft phase area.

6 ACKNOWLEDGEMENT

Laboratory assistance was provided by W. Duvall and T. Strakna, University of Maryland. Partial research support provided by the U.S. Air Force Wright Aeronautical Laboratory is appreciated.

REFERENCES

- (1) PETERSON, M.B. and RAMALINGAM, S., in Fundamentals of Friction and Wear of Materials, ed. D.A. Rigney (ASM, Metals Park, OH, 1981). pp. 331-372.
- (2) CONTE, A.A., ASLE Transactions **26**, 200 (1983).
- (3) LANCASTER, J.K. and MOORHOUSE, P., Tribology International **18**, 139-148 (1985).

PLEASE CHECK THE APPROPRIATE BLOCK BELOW:

-AO # 196-08-1926

☒ 1 copies are being forwarded. Indicate whether Statement A, B, C, D, E, F, or X applies.



DISTRIBUTION STATEMENT A:

APPROVED FOR PUBLIC RELEASE: DISTRIBUTION IS UNLIMITED



DISTRIBUTION STATEMENT B:

DISTRIBUTION AUTHORIZED TO U.S. GOVERNMENT AGENCIES ONLY; (Indicate Reason and Date). OTHER REQUESTS FOR THIS DOCUMENT SHALL BE REFERRED TO (Indicate Controlling DoD Office).



DISTRIBUTION STATEMENT C:

DISTRIBUTION AUTHORIZED TO U.S. GOVERNMENT AGENCIES AND THEIR CONTRACTORS; (Indicate Reason and Date). OTHER REQUESTS FOR THIS DOCUMENT SHALL BE REFERRED TO (Indicate Controlling DoD Office).



DISTRIBUTION STATEMENT D:

DISTRIBUTION AUTHORIZED TO DoD AND U.S. DoD CONTRACTORS ONLY; (Indicate Reason and Date). OTHER REQUESTS SHALL BE REFERRED TO (Indicate Controlling DoD Office).



DISTRIBUTION STATEMENT E:

DISTRIBUTION AUTHORIZED TO DoD COMPONENTS ONLY; (Indicate Reason and Date). OTHER REQUESTS SHALL BE REFERRED TO (Indicate Controlling DoD Office).



DISTRIBUTION STATEMENT F:

FURTHER DISSEMINATION ONLY AS DIRECTED BY (Indicate Controlling DoD Office and Date) or HIGHER DoD AUTHORITY.



DISTRIBUTION STATEMENT X:

DISTRIBUTION AUTHORIZED TO U.S. GOVERNMENT AGENCIES AND PRIVATE INDIVIDUALS OR ENTERPRISES ELIGIBLE TO OBTAIN EXPORT-CONTROLLED TECHNICAL DATA IN ACCORDANCE WITH DoD DIRECTIVE 5230.25, WITHHOLDING OF UNCLASSIFIED TECHNICAL DATA FROM PUBLIC DISCLOSURE, 6 Nov 1984 (Indicate date of determination). CONTROLLING DoD OFFICE IS (Indicate Controlling DoD Office).



This document was previously forwarded to DTIC on _____ (date) and the AD number is _____.



In accordance with provisions of DoD instructions, the document requested is not supplied because:



It will be published at a later date. (Enter approximate date, if known).



Other. (Give Reason)

DoD Directive 5230.24, "Distribution Statements on Technical Documents," 18 Mar 87, contains seven distribution statements, as described briefly above. Technical Documents must be assigned distribution statements.

Joseph A. Burke

Print or Type Name

DSN785-5197

Telephone Number

Joseph A. Burke
Authorized Signature/Date